

Attorney Docket No.: 0209-PA

PATENT

**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

In re Application of:	)	
Michael T. COSTELLO, et al.	:	
	)	Examiner: James C. GOLOBOY
U.S. Application No.: 10/731,600	:	
	)	Group Art Unit: 1797
Filed: December 9, 2003	:	
	)	Confirmation No.: 3499
For: LOW SEDIMENT FRICTION	:	
MODIFIERS	)	August 25, 2009

**PETITION TO WAIVE RE-EXECUTION OF DECLARATION OF NON-COOPERATING INVENTOR UNDER 37 C.F.R. §1.183**

Commissioner for Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450

Sir:

Applicants respectfully petition the Commissioner, under 37 C.F.R. §1.183, to waive the 37 CFR 1.64 and 35 U.S.C. §116 requirement for Dr. Igor Riff, a non-cooperating inventor, to re-execute a Declaration for the above-identified patent application. Applicants authorize the Commissioner to charge a \$130 processing fee under 37 C.F.R. §1.48(a) and a \$400 petition fee under 37 C.F.R. §1.183 to Deposit Account No. 50-1710. Any additional fee required, and any overpayments, should be charged to Deposit Account No. 50-1710.

**A. Dr. Hoey contributed to the conception of the invention and should be added as an inventor.**

While employed by Infineum USA L.P. ("Infineum"), Dr. Michael Costello and Dr. Michael Dennis Hoey worked on an invention entitled "Esteramines as Organic Friction Modifiers." This work was documented by Infineum in a patent memorandum, which names Dr. Costello and Dr. Hoey as "Authors/Contributors." A redacted version of this patent memorandum is submitted with this Petition. Later, Dr. Costello was hired by Chemtura Corporation ("Chemtura"), where he, along with Dr. Igor Riff and Dr. Joseph A. Weaver, Jr., worked in further pursuit of the invention. As a result of the collective efforts of Dr.

Costello, Dr. Riff, Dr. Weaver, and Dr. Hoey, U.S. Patent Application No. 10/731,600 (“the Application”) was filed in the United States Patent and Trademark Office (“USPTO”) on December 9, 2003. Dr. Costello and Dr. Weaver have acknowledged Dr. Hoey’s contribution and, as such, have each re-executed their respective Declarations to reflect Dr. Hoey’s inclusion as an inventor. These re-executed Declarations are being concurrently submitted along with a Request to Correct Inventorship under 35 U.S.C. §116. Dr. Hoey, while working with Dr. Costello at Infineum, contributed to the conception of the invention, and, accordingly, should be added as an inventor.

**B. Dr. Costello, Dr. Weaver, and Chemtura agree that Dr. Hoey is an inventor.**

The original Declaration relating to the Application was executed by Dr. Costello, Dr. Riff, and Dr. Weaver. In addition, each of the inventors assigned their rights in the Application to Crompton Corporation on January 5, 2004. Crompton Corporation changed its name to Chemtura Corporation (“Chemtura”) on July 1, 2005. On July 25, 2008, Michael Dennis Hoey assigned his interest in the invention to Chemtura. Accordingly, Chemtura Corporation is the Assignee of the Application.

Upon learning of Dr. Hoey’s contribution to the invention, Chemtura promptly took the appropriate steps to correct the inventorship of the Application. As indicated above, Chemtura has obtained re-executed Declarations from Dr. Costello and Dr. Weaver, which reflect that Dr. Hoey contributed to the subject matter of the claimed invention. Thus, with the exception of Dr. Riff, all of the inventors agree that Dr. Hoey should be added as an inventor. In addition, Chemtura has obtained Dr. Hoey’s Statement that Inventorship Error Occurred without Deceptive Intention. Further, Chemtura has consented to the correction of inventorship and has executed a Consent to Corrected Inventorship under 37 C.F.R. §1.48(a). Thus, Dr. Costello, Dr. Weaver, and Chemtura agree that Dr. Hoey is an inventor for the Application.

**C. Statement of facts relating to Dr. Riff’s refusal to re-execute the Declaration.**

--Dr. Riff’s employment with Chemtura ended on or about November 30, 2004, which was before Chemtura was aware of Dr. Hoey’s role in the invention.

--Upon learning of Dr. Hoey's role in the invention, Chemtura has contacted Dr. Riff and requested that he re-execute his Declaration to reflect Dr. Hoey's inclusion as an inventor. Chemtura also requested that Dr. Riff speak with Dr. Costello and Dr. Weaver regarding the Application. Dr. Riff refused to do so.

--On May 8, 2009, Chemtura's representative sent a letter, via Certified Mail, Return Receipt Requested, to Dr. Riff. In the May 8, 2009 letter, Chemtura's representative encouraged Dr. Riff to consult with Dr. Costello and Dr. Weaver to verify that Dr. Hoey did, indeed, contribute to the invention. Dr. Riff refused to speak with Dr. Costello concerning this matter.

--The May 8, 2009 letter also requested that Dr. Riff re-execute his Declaration by May 15, 2009.

--The May 8, 2009 letter was sent to 5 Colonial Court, Skillman, NJ, 08558, which was the last known address for Dr. Riff. The May 8, 2009 letter was accompanied by a copy of the application, including the claims, the specification, and the drawings, as originally filed with the USPTO, a copy of the Declaration as originally filed with the USPTO, and a copy of the amended claims that were then before the USPTO. Copies of the May 8, 2009 letter and the enclosures are submitted with this Petition.

--In the May 8, 2009 letter, Dr. Riff was made aware that, if he did not provide a re-executed Declaration, Chemtura would presume that Dr. Riff refused to sign the Declaration and that Chemtura would pursue the correction of inventorship under the USPTO rules relating to uncooperative inventors.

--Dr. Riff received the May 8, 2009 letter and signed the Certified Mail Return Receipt on May 11, 2009. A copy of the Certified Mail Return Receipt is submitted with this Petition.

--In response to the May 8, 2009 letter, Dr. Riff sent a letter dated August 17, 2009 to Chemtura's representative, which clearly reflects his refusal to re-execute his Declaration. A copy of Dr. Riff's August 17, 2009 letter is submitted with this Petition.

**D. Applicants request waiver of the requirement of re-execution of Declaration of Non-Cooperating Inventor, Dr. Riff, under 37 C.F.R. §1.183.**

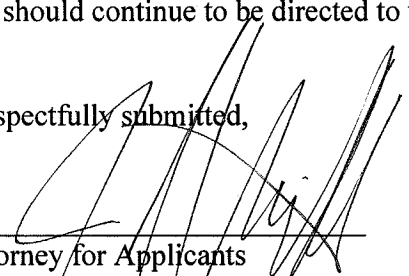
As indicated above, Chemtura, Dr. Hoey, Dr. Costello, and Dr. Weaver agree that Dr. Hoey is an inventor in the Application and should be named as such. Further, Chemtura has

given its consent to correct the inventorship and has taken measures to do so. Dr. Riff has refused to cooperate in the addition of Dr. Hoey as an inventor in the Application, has refused to consult with Dr. Costello and Dr. Weaver to verify Dr. Hoey's inventive contribution, and has refused to re-execute the Declaration required to correct the inventorship of the Application.

Under 37 C.F.R. §1.183, in an extraordinary situation, when justice requires, any requirement of the regulations in this part which is not a requirement of the statutes may be suspended or waived by the Director or the Director's designee, *sua sponte*, or on petition of the interested party, subject to such other requirements as may be imposed. Further, in such circumstances, a petition under 37 CFR 1.183, requesting waiver of the 37 CFR 1.64 and 35 U.S.C. §116 requirement that each of the actual inventors execute the oath or declaration, should be considered *particularly where assignee consent is given to the requested correction*. (See *Manual of Patent Examination Procedure (MPEP)* §201.03). Accordingly, because Dr. Riff refuses to re-execute the Declaration with the corrected inventorship and because Chemtura has consented to the correction of inventorship, Applicants respectfully petition the Commissioner, under 37 C.F.R. §1.183, to waive the requirement of re-execution of a Declaration as it relates Dr. Riff.

Applicants' undersigned attorney may be reached in our Washington, D.C. office by telephone at (202) 625-3500. All correspondence should continue to be directed to the address given below.

Respectfully submitted,



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Attorney for Applicants  
Jeffrey T. Gendzwil  
Registration No. 55,201  
KATTEN MUCHIN ROSENMAN LLP

**Please continue to direct all  
correspondence to:**  
Patent Administrator  
Chemtura Corporation  
199 Benson Road  
Middlebury, CT 06749  
USA



PROPRIETARY INFORMATION  
For Authorized Company Use Only

### SHORT PM FORM

To: Law Technology

Cc:

Michael Hoey and Michael  
Costello

Patent Team Attorney

Patent Team Technologist

Technology Leader/Manager

Patent Team Paralegal

FOR LTC USE

PM # **2001M036**

Received: [REDACTED]

(Attorney Signature)

First Date of Receipt:

**2001**

Working Title: Esteramines as  
Organic Friction Modifiers

#### Invention Title

Esteramines as Organic Friction Modifiers

#### Author(s) / Contributor(s)

Michael Hoey and Michael Costello

#### Idea - Brief summary of the inventive concept highlighting the problem solved and the novel features.

- Background to the concept - Please include date of first written description

We want to claim the use of esteramines as organic friction modifiers in lubricating fluids. Organic Friction Modifiers are used in a number of lubrication fluids, including crankcase oils. In crankcase, the currently used OFM's may be limited in treat, and performance, by instabilities that they cause with other components, such as overbased detergents, or [REDACTED]. We determined that an ester combined with an amine functionality should give good performance in lubricity and should be better in detergent (C9330) stability. We then selected esteramines, a commercial class of products used as an intermediate to make esterquats, as a likely product category. Esteramines are a class of compound that is formed by the reaction of a tertiary hydroxyl substituted amine with an alkyl carboxylic acid, such as a fatty acid. Two examples are TEEMA, which is tallow acid reacted with triethanolamine, and DEEMA, which is tallow acid reacted with methyl diethanolamine. These materials have been tested and have shown improved frictional performance and increased stability with overbased calcium sulfonate detergents when compared to current OFM's such as GMO and ET-2. Another material not tested, but within the scope of this disclosure is the ester product from a fatty acid and an ethoxylated alkyl amine, such as ET-2.

#### Benefit

##### Technical Advantage of Your Idea versus Current Practice

The advantage of these types of compounds lies in the additional flexibility that they would give one in formulating a lubricating oil. The current treat limitations for currently used OFM's is established by the interactions (problems) that are generated by detergent stability and [REDACTED]. These products could also have a commercial advantage depending upon the raw material costs, and they may also have better performance in [REDACTED].

##### Any Data?

Formulas made with TEEMA and DEEMA (obtained from [REDACTED]) showed excellent stability performance and better frictional performance when compared to formulas with GMO and ET-2.

Current practice



**Business Areas:** Crankcase and PTF, 2T, Specialities, Patent Issues (maybe) with Fuel. (PTF, Crankcase, Fuels, 2T, Gear, Other).  
Specify Patent Team if Possible \_\_\_\_\_

Do you need support or the participation of others?      Yes *X*      No

[REDACTED] 2001

**Paralegal will arrange follow-up with Patent Team Attorney, Sr. Technologist and Tech Leader within two weeks.**

2900 K Street NW  
Suite 200  
Washington, DC 20007-5118  
202.625.3500 tel  
202.298.7570 fax

May 8, 2009

**VIA CERTIFIED MAIL**  
**RETURN RECEIPT REQUESTED**

JUSTIN L. KRIEGER  
justin.krieger@kattenlaw.com  
202.625.3858 direct  
202.295.1166 fax

Dr. Igor Riff  
5 Colonial Court  
Skillman, NJ 08558  
U.S.A.

Re: U.S. Patent Application No. 10/731,600  
For: LOW SEDIMENT FRICTION MODIFIERS  
Chemtura Ref. 0209-PA  
Our Ref. 338004-07801

Dear Dr. Riff:

This letter is further to our previous communications regarding the addition of Dr. Michael Dennis Hoey of Infineum USA L.P. ("Infineum") as an inventor of U.S. Patent Application No. 10/731,600 ("the Application"). As we discussed, while at Infineum, Dr. Hoey worked with Michael T. Costello on the subject matter of the Application. Dr. Hoey's contribution has been well-documented and confirmed by Infineum, as indicated in the enclosed Infineum Patent Memorandum. Dr. Costello was then hired by Chemtura, where he worked with you and Joseph A. Weaver, Jr. in further pursuit of the invention. Ultimately, the Application was filed on December 9, 2003 as a result of all of your collective efforts. Because of Dr. Hoey's contributions while working with Dr. Costello at Infineum, Dr. Hoey should properly be named as a co-inventor. Thus, the complete list of inventors for the Application should include you, Dr. Costello, Dr. Weaver and Dr. Hoey.

We understand that you have previously refused to sign the documents required to add Dr. Hoey as a co-inventor, stating that you do not believe Dr. Hoey is a co-inventor. In response, we have asked you to consult with Dr. Costello to confirm that Dr. Hoey did work with Dr. Costello at Infineum and that Dr. Hoey did, indeed, contribute to the invention. We trust that you have confirmed same with Dr. Costello. If not, we ask that you please do so at your earliest convenience. We also note that Dr. Costello and Dr. Weaver agree that Dr. Hoey did contribute to the invention and should properly be named as one of the co-inventors.

With this letter, we have provided a copy of the application as originally filed with the United States Patent and Trademark Office ("USPTO"), which includes the claims, the specification, and the drawings. We have also provided a copy of the original Declaration. In addition, we have provided a copy of the amended claims that are currently before the USPTO.



Dr. Igor Riff  
May 8, 2009  
Page 2

In addition to the originally-filed papers, we have again provided a Combined Declaration for Patent Application and Power of Attorney ("the Declaration") relating to the Application for your execution. The Declaration lists the following inventors:

Dr. Igor Riff;  
Dr. Michael T. Costello;  
Dr. Joseph A. Weaver, Jr.; and  
Dr. Michael Dennis Hoey.

We ask that you kindly review the originally-filed application papers and the currently pending claims and execute the Declaration and return it to us by **May 15, 2009** so that we may file same with the USPTO. If we do not hear from you by May 15, 2009, we will presume that you refuse to sign the Declaration and we will have no choice but to correct the inventorship under the USPTO rules relating to uncooperative inventors.

We look forward to hearing from you in the near future. Should you have any questions, please do not hesitate to contact us.

Sincerely,

Justin L. Krieger

JLK:jm

cc: Jaimes Sher (Chemtura)

encl: Patent Memorandum  
Original Application as filed with USPTO  
Original Declaration as filed with USPTO  
Combined Declaration for Patent Application and Power of Attorney  
Amended Claims currently before the USPTO

LOW SEDIMENT FRICTION MODIFIERS

BACKGROUND OF THE INVENTION

1. Technical Field

The present invention relates to friction modifiers for lubricant oils used, for example, in engines for motorized vehicles.

2. Description of the Related Art

Friction modifiers for enhancing the lubricity of lubricant oils are known. Typical friction modifiers include esters of glycerol and fatty acids, overbased carboxylates, and oxidized petroleum fractions. Also known is the use of detergent additives such as overbased calcium sulfonates. However, a common problem which occurs when aging the lubricant oil is the formation of sediment.

During storage, especially under warm temperature conditions, sedimentation occurs in stocks of lubricant oils containing friction modifiers and detergent additive. In a storage tank containing many thousands of gallons of lubricant oil fluid, a sedimentation rate of a few percent can correspond to a thousand or more gallons of bottom sediment, which is clearly undesirable. What is needed is an additive for lubricant oils which provides a friction modifier and detergent, and which is characterized by a low sedimentation rate.

SUMMARY

An additive mixture for lubricant oils is provided herein. The additive mixture comprises: (a) an overbased alkaline earth metal sulfonate; and, (b) at least one friction modifier selected from the group consisting of an overbased alkaline earth carboxylate, the reaction

product of an alkanolamine with a fatty acid or a fatty acid ester, the reaction product of thiodiglycol with a fatty acid or a fatty ester and the reaction product of a dialkylene glycol with a fatty acid or a fatty ester.

The additive mixture provides a lubricant oil composition having improved lubricity and exhibits less sedimentation.

#### BRIEF DESCRIPTION OF THE DRAWINGS

Various embodiments are described below with reference to the drawings wherein:

FIG. 1 is a graph illustrating the sedimentation results of Example 75; and,

FIG. 2 is a graph illustrating the friction test results of Example 76.

#### DETAILED DESCRIPTION OF PREFERRED EMBODIMENT(S)

Lubricating oil compositions used to lubricate the moving parts of engines, especially internal combustion engines, contain base oil of lubricating viscosity, or a mixture of such oils, and additives used to improve the performance characteristics of the oil. For example, additives are used to improve detergency, reduce engine wear, to provide stability against heat and oxidation, to reduce oil consumption, to inhibit corrosion, to act as a dispersant, and reduce friction loss. Some additives provide multiple benefits, such as a dispersant/viscosity modifier. Other additives, while improving one characteristic of the lubricating oil, have an adverse affect on other characteristics. Thus to provide a lubricating oil having optimal overall performance, it is necessary to characterize and understand all the effects of the various additives available, and carefully balance the additive content of the lubricant.

The present invention is a low sediment additive mixture for lubricant oils which comprises an overbased alkaline earth metal sulfonate with a selected friction modifier.

The additive mixture can be added to any type of lubricant oil including metal cutting and drawing oils, although motor oils are preferred. The lubricant oils can be natural or synthetic. Natural oils include petroleum oils, solvent-treated, acid treated or hydrotreated mineral oils, coal oil and shale oil. The lubricant oil can include saturated, olefinic unsaturated and aromatic components. Furthermore, the lubricant oil can include hydrocarbon and/or non-hydrocarbon components such as fatty acids and fatty acid esters.

Overbased alkaline earth sulfonates are detergent compounds prepared by neutralizing a sulfonic acid with an excess of alkaline earth metal base (e.g., the hydroxides of magnesium, calcium or barium) so as to produce an overbased alkaline earth metal sulfonate with a total base number ("TBN") of greater than zero. The TBN is the amount of acid needed to neutralize all of the alkalinity of the overbased material, and can be determined according to ASTM D 2896. A composition with a TBN of about 100 or less is considered to be a "low overbased material." A TBN of about 100 to 300 is characterized as "moderate overbasing." A composition with a TBN of over 300 is considered to be a "highly overbased" material. The preferred overbased alkaline earth metal sulfonate is overbased calcium sulfonate having a TBN of above about 250, preferably above about 350, and more preferably 400 or higher.

In one aspect of the invention it has been found that amorphous overbased calcium sulfonate is superior to crystalline calcium overbased sulfonate in terms of undesired sedimentation. An amorphous overbased calcium sulfonate suitable for use in the present inventor in commercially available from Crompton Corporation of Middlebury CT under the designation Calcinat<sup>TM</sup> C-400 CLR. The amorphous overbased calcium sulfonate has a particle size of less than about 30 nm. Crystalline overbased calcium sulfonates have a particle size above 30 nm, preferably 50-500 nm and more preferably 50-100 nm. Calcinat<sup>TM</sup> C-300CS, available from Crompton Corporation, is an example of a crystalline overbased calcium

sulfonate. A method for preparing highly overbased calcium sulfonate is described in U.S. Patent No. 6,444,625 B1, which is herein incorporated by reference.

In another aspect of the invention it has been found that certain friction modifiers, when combined with an overbased alkaline earth sulfonate, exhibit lower sedimentation properties.

5 The lubricant composition also comprises a friction modifier to reduce the coefficient of friction. The friction modifier and overbased calcium sulfonate can be combined and packaged with other additives such as antioxidants, dispersants and/or defoamers, or other types of additives such as mentioned above.

0 Various friction modifiers can be used in the additive mixture. Such friction modifiers include glycerol monoesters, overbased carboxylates, overbased tall oil fatty acids, the reaction product of an alkanolamine (e.g., triethanolamine ("TEA") or diethanolamine) or a glycol (e.g., thiodiglycol, diethylene glycol) with a fatty acid or fatty ester, oxygenated petroleum fractions, alkoxyated alkylamine and the reaction products of glycols with fatty esters.

5 Glycerol monoesters useful as friction modifiers include, for example, glycerol esters of saturated or unsaturated C<sub>8</sub> to C<sub>20</sub> fatty acids such as glycerol monopalmitate, glycerol monostearate, glycerol monooleate, and the like.

0 Overbased carboxylates are known and are generally prepared by reacting an acidic material, normally an acidic gas such as SO<sub>2</sub> or CO<sub>2</sub>, and most commonly carbon dioxide, with a mixture comprising a carboxylic acid and a stoichiometric excess of an alkaline base metal compound in a reaction medium, preferably with a promoter. The base metal is preferably an alkaline earth metal such as magnesium, calcium or barium in the form of an oxide or hydroxide. The carboxylic acid is preferably a saturated or unsaturated carboxylic acid having from about 8 to about 30 carbon atoms. Useful carboxylic acids include, but are not limited to, caprylic acid, capric acid, lauric acid, myristic acid, myristoleic acid, decanoic acid, dodecanoic acid, 5 pentadecanoic acid, palmitic acid, palmitoleic acid, margaric acid, stearic acid, 12-

hydroxystearic acid, oleic acid, ricinoleic acid, linoleic acid, arachidic acid, gadoleic acid, eicosadienoic acid, behenic acid, erucic acid, mixtures of any of these acids or their reactive equivalent.

5 Other suitable overbased carboxylates include overbased calcium tallate and overbased barium tallate.

A reaction product of TEA and fatty ester suitable for use as a friction modifier is the reaction product of TEA and methyl oleate. Other suitable friction modifiers include the reaction products of TEA with, for example, oleic acid, ricinoleic acid, isostearic acid, erucic acid, tall oil fatty acid (TOFA), mixed oleic/stearic acids, and iso-oleic acid.

0 Oxygenated petroleum fractions are known. Petroleum oxidates and methods for making them are disclosed in U.S. Patent No. 5,439,602, which is herein incorporated by reference.

Also suitable for use as friction modifiers are the reaction products of thiodiglycol with fatty acids or fatty esters (e.g., oleic acid, methyl oleate, etc.), and the reaction products of a dialkylene glycol (e.g., diethylene glycol, dipropylene glycol, etc.) with a fatty acid or fatty ester  
5 (e.g., oleic acid, methyl oleate, etc.).

In another aspect of the invention it has been found that overbased alkaline earth sulfonate (especially crystalline overbased calcium sulfonate) provides a lubricant oil composition with greater lubricity (i.e., lower coefficient of friction). Particularly preferred as a friction modifier are the reaction products of TEA with fatty acids or fatty esters for both  
0 excellent lubricity and low sedimentation. A preferred crystalline overbased calcium sulfonate is available from Crompton Corp. under the designation Calcinate C300CS and has a particle size of about 50-500 nm. Preferred particle size for a crystalline overbased calcium sulfonate ranges from about 50 nm to about 100 nm.

The following Examples illustrate features of the invention. In one type of experiment,  
5 illustrated below in Examples 1-30 and 59-75, lubricant oils containing a selected overbased

calcium sulfonate and a selected friction modifier were tested for sedimentation. In another type of experiment, exemplified in Examples 31-58 and 76, the lubricant oils containing combinations of selected overbased calcium sulfonates and friction modifiers were tested for friction reducing characteristics.

### Examples 1-8

These examples are directed to a study of the sedimentation characteristics of various blends of overbased calcium sulfonate and selected friction modifiers. Two types of overbased calcium sulfonates were individually tested. Calcinate™ C300CS is a crystalline overbased calcium sulfonate having TBN of about 300 and a particle size of from about 50nm to about 100 nm, and is available from Crompton Corporation. Calcinate™ C400CLR is an amorphous overbased calcium sulfonate available from Crompton Corporation having TBN of about 400 and a particle size of no more than about 30 nm. Overbased calcium sulfonates were present in 10% concentrations. The friction modifier included glycerol mono oleate ("GMO"). Polyisobutylene succinic anhydride ("PIBSA") having a molecular weight of about 1,050, was present in Examples 3, 4, and 7 as a dispersant. The friction modifier was present in 1% concentrations when employed. The lubricant oil stock was a severely hydrotreated naphthenic oil available from Ergon, Inc. under the designation Hyprene H100. Hyprene H100 is characterized by an API gravity (60°F) of 24.6, a pour point of -50°F, and an aniline point of 163°F. The respective overbased calcium sulfonate and friction modifier were combined with the diluent lubricant stock and placed in a 100 ml centrifuge tube in a 70°C oven for a period of 12 weeks. The sediment formed was measured at one week intervals and recorded as percentages. In examples 1 and 5, no friction modifiers were employed. It can be seen that use of the amorphous overbased calcium sulfonate Calcinate C400CLR (Examples 5-8) reduces sedimentation dramatically. It is believed that the reduced sedimentation of the amorphous variety of overbased calcium sulfonate is related to its smaller particle size.

The results of these experiments are set forth in Table 1.

The sediments formed in the Calcinate™ C300CS Examples 1 to 4 were analyzed by FTIR by removing an aliquot of sediment from the bottom layer with a pipette. In all sediments



there was a strong peak at  $881\text{ cm}^{-1}$ , which is an indication of  $\text{CaCO}_3$  in the calcite form. There were no peaks observed from sulfonate, which did not precipitate with the calcite.

TABLE I

Example	1	2	3	4	5	6	7	8
C300CS	10%	10%	10%	10%	---	---	---	---
C400CLR	---	---	---	---	10%	10%	10%	10%
PIBSA	---		1%	1%	---	---	1%	1%
GMO	---	1%	---	1%	---	1%	---	1%
Hyprene H100	90%	89%	89%	88%	90%	89%	89%	88%
% Sediment, 70°C @ Week								
1	0.01	0.35	0.55	0.35	0	0	0	0
2	0.35	0.25	0.50	0.30	0	0	0	0
3	0.30	0.30	0.50	0.30	0	0	0	0
4	0.30	0.30	0.65	0.25	0	0	0	0
5	0.25	0.25	0.50	0.30	0	0	0	0
6	0.25	0.25	0.30	0.25	0.001	0.002	0.001	0.002
7	0.30	0.25	0.40	0.25	0.002	0.002	0.001	0.002
8	0.25	0.25	0.40	0.25	0.002	0.002	0.001	0.005
9	0.25	0.25	0.35	0.25	0.002	0.002	0.002	0.005
10	0.25	0.25	0.40	0.25	0.002	0.002	0.002	0.005
11	0.25	0.25	0.35	0.25	0.002	0.002	0.005	0.010
12	0.25	0.25	0.35	0.25	0.002	0.002	0.005	0.010
Final centrifuge	0.25	0.20	0.35	0.25	0.001	0.002	0.002	0.005

### Examples 9 to 16

Sedimentation experiments were conducted in the same manner as in Examples 1-8 except that the friction modifiers tested were an oxidized petroleum fraction available under the designation Alox 165L from Lubrizol, an overbased calcium oleate, designated herein as “OCO”, and GMO. The friction modifiers, when employed, were each present in 0.5% concentration. Examples 10 and 14 did not employ any friction modifiers.

As can be seen from the results the amorphous overbased calcium sulfonate Calcinat<sup>TM</sup> C400CLR (Examples 13-16) was characterized by very low sedimentation percentages (typically about 0.001 to about 0.005) as opposed to the sedimentation percentages (0.11 to 0.50) for the Calcinat<sup>TM</sup> C300CS (Examples 9-12).

The results of these experiments are set forth in Table 2.

TABLE 2

Example	9	10	11	12	13	14	15	16
C300CS	10%	10%	10%	10%	--	--	--	--
C400CLR	--	--	--	--	10%	10%	10%	10%
Alox 1652	0.5%	--	--	--	0.5%	--	--	--
OCO	--	--	0.5%	--	--	--	0.5%	--
GMO	--	--	--	0.5%	--	--	--	0.5%
Hyprene H100	89.5%	90	89.5	89.5	89.5	90	89.5	89.5
% Sediment, 70°C @ Week								
1	0.50	0.40	0.02	0.40	0.001	0.001	0.001	0.001
2	0.40	0.35	0.03	0.30	0.001	0.001	0.001	0.001
3	0.35	0.30	0.02	0.30	0.001	0.001	0.001	0.001
4	0.35	0.30	0.05	0.30	0.001	0.001	0.001	0.001
5	0.35	0.30	0.11	0.30	0.001	0.001	0.001	0.001
6	0.30	0.25	0.20	0.25	0.001	0.001	0.001	0.002
7	0.30	0.25	0.40	0.25	0.005	0.002	0.002	0.002
8	0.30	0.25	0.20	0.25	0.005	0.005	0.002	0.005
9	0.30	0.25	0.20	0.25	0.005	0.005	0.002	0.005
10	0.30	0.25	0.20	0.25	0.005	0.005	0.002	0.005
11	0.30	0.25	0.25	0.25	0.005	0.005	0.005	0.005
12	0.30	0.25	0.25	0.25	0.005	0.005	0.005	0.005
Final centrifuge	0.30	0.25	0.20	0.25	0.005	0.002	0.002	0.002

### Examples 17 to 28

Sedimentation experiments were conducted in a manner similar to Examples 9 to 16, except that the friction modifiers tested were OCO (overbased calcium oleate), Alox 302 (oxygenated petroleum fraction), an overbased barium tallate having 3% -10% barium and designated herein as "OBT", an overbased calcium tallate having 4%-10% calcium and designated herein as "OCT", and GMO (glycerol mono oleate). Examples 22 and 28 did not employ any friction modifier.

As can be seen, the amorphous overbased calcium sulfonate Calcinat<sup>TM</sup> C400CLR (Example 23-28) is characterized by much lower percentages of sedimentation as opposed to the sedimentation percentages for the crystalline overbased calcium sulfonate Calcinat<sup>TM</sup> C300CS (Examples 17-22).

The results of these experiments are set forth in Table 3

TABLE 3

Example	17	18	19	20	21	22	23	24	25	26	27	28
C300CS	10%	10%	10%	10%	10%	10%	---	---	---	---	---	---
C400CLR	---	---	---	---	---	---	10%	10%	10%	10%	10%	10%
OCO	0.5%	---	---	---	---	---	0.5%	---	---	---	---	---
Alox 302	---	0.5%	---	---	---	---	---	0.5%	---	---	---	---
OBT	---	---	0.5%	---	---	---	---	---	0.5%	---	---	---
OCT	---	---	---	0.5%	---	---	---	---	---	0.5%	---	---
GMO	---	---	---	---	0.5%	---	---	---	---	---	0.5%	---
Hyprene	89.5%	89.5	89.5	89.5	89.5	90	89.5	89.5	89.5	89.5	89.5	90
% Sediment 70°C @ Week												
1	0.001	0.001	0	0	0.45	0.01	0	0	0	0	0	0
2	0.002	0.35	0.01	0.01	0.35	0.25	0	0	0	0	0	0
3	0.030	0.40	0.02	0.04	0.35	0.25	0	0	0.001	0.001	0.001	0.001
4	0.030	0.35	0.05	0.05	0.30	0.25	0	0.001	0.001	0.001	0.001	0.001
5	0.030	0.30	0.03	0.075	0.25	0.25	0.001	0.001	0.002	0.002	0.001	0.001
6	0.030	0.40	0.04	0.10	0.25	0.25	0.001	0.001	0.001	0.001	0.001	0.001
7	0.040	0.35	0.04	0.10	0.25	0.30	0.001	0.001	0.002	0.002	0.001	0.001
8	0.040	0.35	0.05	0.15	0.25	0.30	0.001	0.002	0.002	0.002	0.001	0.002
9	0.050	0.30	0.05	0.15	0.25	0.20	0.001	0.002	0.002	0.002	0.001	0.002
10	0.050	0.30	0.05	0.15	0.25	0.25	0.001	0.002	0.002	0.002	0.001	0.002
11	0.070	0.30	0.05	0.15	0.25	0.25	0.001	0.002	0.002	0.002	0.002	0.002
12	0.070	0.30	0.05	0.15	0.25	0.25	0.001	0.001	0.002	0.002	0.002	0.002

#### Examples 29 to 30

Sedimentation experiments were conducted in the same manner as in the previous examples except that the friction modifiers included 0.5% of a reaction product of TEA and tall oil fatty acid resulting in a trioleate ester, which is designated herein as "TEA-T".

The results set forth in Table 4 illustrate that the amorphous overbased calcium sulfonate Calcinat<sup>TM</sup> C400CLR was (Examples 31, 32) was characterized by much lower sedimentation percentages than the sedimentation percentages for crystalline overbased calcium sulfonate Calcinat<sup>TM</sup> C300CS. But also the use of TEA-T as a friction modifier with Calcinat<sup>TM</sup> C300CS surprisingly provided a lubricant blend with much lower sedimentation than that employing the GMO/ET-2 friction modifier.

**TABLE 4**

Example	29	30
C300 CS	10%	---
C400 CLR	---	10%
TEA-T	0.5%	0.5%
GMO	---	---
Hyprene H100	89.5%	89.5%
Total % Sediment @ 70°C Week:		
1	0.001	0
2	0.010	0.001
3	0.020	0.001
4	0.020	0.001
5	0.020	0.002
6	0.030	0.001
7	0.040	0.002
8	0.050	0.002
9	0.050	0.002
10	0.050	0.002



### Examples 31-44

These Examples are directed to the friction reducing characteristics of the lubricant oil blends. The coefficient of friction for the selected blends was measured with a Cameron-Plint

TE-77 high frequency friction test. The conditions are set forth below

Upper specimen 6mm X 16mm Dowel EN 1A (BS 1804 Part 1 Grade 1, 220M07)  
Lower specimen Flat hardened ground NSOH BO1 Gauge Plate (RC 60/0.4  $\mu\text{m}$ )  
Frequency 5 Hz  
Load (N) 50-100  
Amplitude 15 mm

#### **Temperature Profile for Cameron Plint Test**

Stage	Load (N)	Ramp Time (min)	Temperature °C	Dwell Time (min)
1	0	10	35	5
2	50	10	50	5
3	100	60	165	

In Examples 33 to 48 Calcinat<sup>TM</sup> C300CS and Calcinat<sup>TM</sup> C400CLR are compared on the basis of equal weight..

TABLE 5

Example	31	32	33	34	35	36	37	38	39	40	41	42	43	44
C300CS	10%	10%	10%	10%	10%	10%	10%	-	-	-	-	-	-	-
C400CLR	-	-	-	-	-	-	-	10%	10%	10%	10%	10%	10%	10%
OCO	0.5%	-	-	-	-	-	-	0.5%	-	-	-	-	-	-
Alox 165L	-	0.5%	-	-	-	-	-	-	0.5%	-	-	-	-	-
OBT	-	-	0.5%	-	-	-	-	-	-	0.5%	-	-	-	-
OCT	-	-	-	0.5%	-	-	-	-	-	-	0.5%	-	-	-
GMO	-	-	-	-	0.5%	-	-	-	-	-	-	0.5%	-	-
Tea-T	-	-	-	-	-	-	0.5%	-	-	-	-	-	-	0.5%
Dil Oil	89.5%	89.5%	89.5%	89.5%	89.5%	90%	89.5%	89.5%	89.5%	89.5%	89.5%	89.5%	90%	89.5%
CoF@60	0.1250	0.1125	0.1275	0.1125	0.1000	0.1267	0.1025	0.1325	0.1090	0.1060	0.1100	0.0960	0.1390	0.1210
70	0.1150	0.1050	0.1175	0.1060	0.0960	0.1258	0.0975	0.1375	0.1060	0.1125	0.1100	0.0910	0.1450	0.1265
80	0.1100	0.1025	0.1100	0.1020	0.0940	0.1222	0.0950	0.1390	0.1060	0.1150	0.1100	0.0900	0.1440	0.1270
90	0.0950	0.0950	0.1000	0.0975	0.0900	0.1160	0.0890	0.1375	0.1040	0.1060	0.1060	0.0875	0.1440	0.1270
100	0.0875	0.0860	0.0925	0.0910	0.0860	0.1115	0.0860	0.1375	0.1020	0.0980	0.1025	0.0850	0.1425	0.1265
110	0.0825	0.0825	0.0860	0.0890	0.0825	0.1013	0.0820	0.1375	0.1000	0.0910	0.1025	0.0840	0.1425	0.1245
120	0.0825	0.0790	0.0825	0.0850	0.0790	0.0945	0.0740	0.1325	0.0975	0.0840	0.1000	0.0810	0.1390	0.1213
130	0.0825	0.0775	0.0790	0.0810	0.0760	0.0908	0.0690	0.1300	0.0960	0.0760	0.0980	0.0800	0.1150	0.1163
140	0.0850	0.0760	0.0775	0.0800	0.0760	0.0883	0.0660	0.1325	0.0940	0.0760	0.0950	0.0790	0.1075	0.1150
150	0.0875	0.0800	0.0800	0.0790	0.0825	0.0867	0.0700	0.1340	0.0925	0.0660	0.0930	0.0775	0.1125	0.1125
160	0.0875	0.0800	0.0825	0.0800	0.0820	0.0858	0.0720	0.1275	0.0950	0.0590	0.0900	0.0750	0.1175	0.1090
Average	0.0945	0.0887	0.0941	0.0912	0.0858	0.1045	0.0821	0.1344	0.1002	0.0900	0.1015	0.0842	0.1317	0.1206

#### Examples 45-58

These Examples were conducted in a manner similar to those of Examples 33 to 49 except that Calciate™ C300CS and Calciate™ C400CLR were compared on the basis of equal TBN rather than equal weight. The results are set forth in Table 6.

TABLE 6

Example	45	46	47	48	49	50	51	52	53	54	55	56	57	58
C300CS	10%	10%	10%	10%	10%	10%	10%	-	-	-	-	-	-	-
C400CLR	-	-	-	-	-	-	-	10%	10%	10%	10%	10%	10%	10%
OCO	0.5%	-	-	-	-	-	-	0.5%	-	-	-	-	-	-
Alox 165L	-	0.5%	-	-	-	-	-	-	0.5%	-	-	-	-	-
OBT	-	-	0.5%	-	-	-	-	-	-	0.5%	-	-	-	-
OCT	-	-	-	0.5%	-	-	-	-	-	-	0.5%	-	-	-
GMO	-	-	-	-	0.5%	-	-	-	-	-	-	0.5%	-	-
TEA-T	-	-	-	-	-	-	0.5%	-	-	-	-	-	-	0.5%
Dil Oil	89.5%	89.5%	89.5%	89.5%	89.5%	90%	89.5%	89.5%	89.5%	89.5%	89.5%	89.5%	90%	89.5%
CoF@60	0.1250	0.1125	0.1275	0.1125	0.1000	0.1267	0.1025	0.1325	0.1090	0.1060	0.1100	0.0960	0.1390	0.1210
70	0.1150	0.1050	0.1175	0.1060	0.0960	0.1258	0.0975	0.1375	0.1060	0.1125	0.1100	0.0910	0.1450	0.1265
80	0.1100	0.1025	0.1100	0.1020	0.0940	0.1222	0.0950	0.1390	0.1060	0.1150	0.1100	0.0900	0.1440	0.1270
90	0.0950	0.0950	0.1000	0.0975	0.0900	0.1160	0.0890	0.1375	0.1040	0.1060	0.1060	0.0875	0.1440	0.1270
100	0.0875	0.0860	0.0925	0.0910	0.0860	0.1115	0.0860	0.1375	0.1020	0.0980	0.1025	0.0850	0.1425	0.1265
110	0.0825	0.0825	0.0860	0.0890	0.0825	0.1013	0.0820	0.1375	0.1000	0.0910	0.1025	0.0840	0.1425	0.1245
120	0.0825	0.0790	0.0825	0.0850	0.0790	0.0945	0.0740	0.1325	0.0975	0.0840	0.1000	0.0810	0.1390	0.1213
130	0.0825	0.0775	0.0790	0.0810	0.0760	0.0908	0.0690	0.1300	0.0960	0.0760	0.0980	0.0800	0.1150	0.1163
140	0.0850	0.0760	0.0775	0.0800	0.0760	0.0883	0.0660	0.1325	0.0940	0.0760	0.0950	0.0790	0.1075	0.1150
150	0.0875	0.0800	0.0800	0.0790	0.0825	0.0867	0.0700	0.1340	0.0925	0.0660	0.0930	0.0775	0.1125	0.1125
160	0.0875	0.0800	0.0825	0.0800	0.0820	0.0858	0.0720	0.1275	0.0950	0.0590	0.0900	0.0750	0.1175	0.1090
Average	0.0945	0.0887	0.0941	0.0912	0.0858	0.1045	0.0821	0.1344	0.1002	0.0900	0.1015	0.0842	0.1317	0.1206

#### Examples 59-74

A study was made of various friction modifiers to determine sedimentation at 12 weeks . The Experiments were conducted by blending crystalline overbased calcium sulfonate Calcinat C300CS ( 10 wt%) with Hyprene H100 lubricant oil stock, and 0.5 % of the selected friction modifier. The composition was then placed in a 70°C oven in a 100 ml centrifuge tube and the sediment formed was measured at one week intervals. The sediments formed ranged from flocculant white/gray material to black solids, although no attempts were made to separately quantify the different types observed. Rather, only the total amount of sediment observed in the centrifuge was recorded so that simple comparisons could be made. The total amount of sediments formed at the end of the 12 week period are reported in Table 7. For comparison, the blend of Calcinat C300CS plus lubricant oil but without a friction modifier was conducted to provide a baseline comparison and was recorded at 0.25%. The mixed oleic/stearic glycol is available from Arizona Chemical Co. under the designation Uniflex 1803. The friction modifiers of Examples 65-74 are the reaction products of the specified compounds.

As can be seen the use of friction modifiers comprising the reaction product of TEA with methyl oleate, tall oil fatty acid, oleic acid, isostearic acid and mixed oleic/stearic acids resulted in low sedimentation as compared with the baseline level of 0.25%. Also, the use of the reaction product of thiodiglycol and methyl oleate resulted in low sedimentation. Overbased alkaline earth carboxylates also provided low sedimentation.

Table 7

Example	Friction Modifier	Sediment (12 Weeks)
59	Glycerol mono oleate	0.25%
60	Overbased calcium carboxylate (OCO)	0.25%
61	Overbased barium carboxylate (OBT)	0.05%
62	Overbased calcium carboxylate (OCT)	0.15%
63	Oxidized petrolatum (Alox 165L)	0.30%
64	Mixed oleic/stearic glycol (Uniflex 1803)	0.25%
65	TEA + methyl oleate	0.05%
66	TEA + tall oil fatty acid	0.06%
67	TEA + oleic acid	0.05%
68	TEA + ricinoleic acid	5.10%
69	TEA + isostearic acid	0.15%
70	TEA + erucic acid	0.35%
71	TEA + mixed oleic/stearic acids	0.20%
72	TEA + iso-oleic acid	1.10%
73	Thiodiglycol + methyl oleate	0.20%
74	Diethylene glycol + methyl oleate	4.20%

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Example 75

The sedimentation tests of Examples 59 to 74 were extended past the 12 week period to determine the nature of failure mode of separation. The results of selected friction modifiers are shown in FIG. 1. A comparison is made with friction modifier produced by the reaction of TEA with a monomeric fatty acid distillate obtained from the dimerization of tall oil fatty acid, such as a mixture of branched and straight chain fatty acids available from Arizona Chemical Co. under the designation Century™ D-1. Also shown in FIG. 1 are the sedimentation test results of a friction modifier produced by the

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reaction of TEA with a mixture of C18 mon-unsaturated mostly methyl branched fatty acids, available from Arizona Chemical Co. under the designation Century™ D1164.

The results show an initial induction period of very low sedimentation followed by a catastrophic failure the sediment completely precipitates in a one week period forming a distinct layer. The layer then continues to compact over the next 2 - 4 weeks and reduces the sediment volume, as can be seen from the TEA + ricinoleic acid, or diethylene glycol + methyl oleate tests. In a second mode the sediment layer forms more slowly after the induction period, but a flocculant material is observed in the supernatant liquid. While the sediment layer does not settle any further, the floc in the supernatant liquid appears to separate into a distinct layer which causes the level of sediment to increase over time, as can be seen from the tests involving TEA + TOFA, TEA + oleic acid, and TEA + iso-oleic acid. There does not appear to be any correlation between the failure mode and the structure of the friction modifier used. However, while not wishing to be bound to any specific theory, it is suggested that the formation of a more dense sediment would more completely separate in the catastrophic failure mode whereas a lighter precipitate would tend to flocculate and settle more slowly after failure.

#### Example 76

Friction tests were performed on selected combinations of overbased calcium sulfonate and friction modifiers of Example 75 in accordance with the testing method set forth in Examples 31-58. The results are illustrated in FIG.2.

It can readily be appreciated that storage of a lubricant oil composition in a vessel over an extended period of time can be accompanied by much less sedimentation by adding to a lubricant stock a mixture including an overbased alkaline earth metal sulfonate, such as overbased calcium sulfonate, and a friction modifier in accordance with the invention.

#### Example 77

This Example illustrates the preparation of the reaction product of TEA with tall oil fatty acid.

A 250mL 3-neck round-bottom flask equipped with an agitator, thermocouple and distillation condenser was charged with 0.062 mol triethanol amine, 0.185 tall oil fatty acid and 0.003 mol para-toluene sulfonic acid catalyst. The system was purged with nitrogen for 3-4 minutes, then the nitrogen flow was turned down to a minimum. The system was agitated and heated to 160°C. Once at this temperature, the nitrogen flow was increased to facilitate the removal of water. Conditions were held for 5 hours after which time IR analysis showed the acid carbonyl absorbance (1712 cm<sup>-1</sup>) to be absent. The reaction was cooled to 100°C and the catalyst was neutralized with 0.70 grams of a Hitec 611 (a 300 TBN detergent). The reaction was filtered to remove solids. The product obtained, was 62 grams of light amber, low viscosity liquid.



### Example 78

This example illustrates the synthesis of a reaction product of diethylene glycol with methyl oleate to obtain ethylene glycol dioleate.

25 Parts of diethylene glycol and 139.7 parts of methyl oleate were combined in a 3-neck round bottom flask. The mixture was heated to 100°C with stirring under a nitrogen sweep. At 100°C, 0.26 parts of dibutyl tin diacetate catalyst was added and the mixture was then heated to 180°C. After 4.33 hours the system was cooled to 100°C and 1.1 parts of Filtrol 20x acid clay was added to neutralize the catalyst. The system was stirred for 15 minutes and the contents of the flask were then filtered with vacuum. The final product was 136.43 parts of low viscosity dark yellow fluid.

While the above description contains many specifics, these specifics should not be construed as limitations of the invention, but merely as exemplifications of preferred embodiments thereof. Those skilled in the art will envision many other embodiments within the scope and spirit of the invention as defined by the claims appended hereto.

**WHAT IS CLAIMED IS:**

1. An additive mixture for lubricant oils which comprises:

a) an overbased alkaline earth metal sulfonate; and,

b) at least one friction modifier selected from the group consisting of a

polyalkylene succinic anhydride, an overbased alkaline earth carboxylate, the reaction  
product of an alkanolamine with a fatty acid or a fatty ester, the reaction product of  
thiodiglycol with a fatty acid or a fatty ester and the reaction product of a dialkylene  
glycol with a fatty acid or a fatty ester.

2. The additive mixture of claim 1 wherein the overbased alkaline earth metal  
sulfonate is overbased calcium sulfonate.

3. The additive mixture of claim 2 wherein the overbased calcium sulfonate is an  
amorphous overbased calcium sulfonate having a particle size of no more than about  
30 nm.

4. The additive mixture of claim 2 wherein the overbased calcium sulfonate is a  
crystalline overbased calcium sulfonate having a particle size of from at least about 50  
nm to about 100 nm.

5. The additive mixture of claim 1 wherein the friction modifier comprises  
polyisobutylene succinic anhydride.

6. The additive mixture of claim 1 wherein the friction modifier comprises calcium carboxylate.

5 7. The additive mixture of claim 1 wherein the friction modifier comprises barium carboxylate.

8. The additive mixture of claim 1 wherein the friction modifier comprises the reaction product of triethanolamine with a fatty acid or fatty acid ester.

10 9. The additive mixture of claim 8 wherein the friction modifier comprises the reaction product of triethanolamine with one or more of a fatty compound selected from the group consisting of methyl oleate, tall oil fatty acid, oleic acid, ricinoleic acid, isostearic acid, erucic acid, mixed oleic acid/stearic acid and iso-oleic acid.

15 10. The additive mixture of claim 1 wherein the friction modifier comprises the reaction product of thiodiglycol with methyl oleate.

11. The additive mixture of claim 1 wherein the friction modifier comprises the reaction product of a diethylene glycol with methyl oleate.

20 12. A lubricant composition comprising:

a) a lubricant oil stock

b) an overbased alkaline earth metal sulfonate; and,

c) at least one friction modifier selected from the group consisting of a polyalkylene succinic anhydride, an overbased alkaline earth carboxylate, the reaction product of an alkanolamine with a fatty acid or a fatty ester, the reaction product of thiodiglycol with a fatty acid or a fatty ester and the reaction product of a dialkylene glycol with a fatty acid or a fatty ester.

13. The lubricant composition of claim 12 wherein the overbased alkaline earth sulfonate is an amorphous overbased calcium sulfonate having a particle size of no more than about 30 nm.

14. The lubricant composition of claim 12 wherein the overbased alkaline earth sulfonate is a crystalline overbased calcium sulfonate having a particle size of from at least about 30 nm to about 50 nm.

15. The lubricant composition of claim 12 wherein the friction modifier comprises the reaction product of triethanolamine with a fatty acid or fatty acid ester.

16. The lubricant composition of claim 15 wherein the friction modifier comprises lubricant composition.

17. The lubricant composition of claim 12 wherein the friction modifier comprises the reaction product of thiodiglycol with methyl oleate.

18. The lubricant composition of claim 12 wherein the friction modifier comprises the reaction product of diethylene glycol with methyl oleate.

19. A method for storing a lubricant composition comprising the steps of :

5 a) combining with a lubricant stock an additive mixture including

i) an overbased alkaline earth metal sulfonate; and,

ii) at least one friction modifier selected from the group consisting of a polyalkylene succinic anhydride, an overbased alkaline earth carboxylate, the reaction product of an alkanolamine with a fatty acid or a fatty acid ester, the reaction product of thiodiglycol with a fatty acid or a fatty ester and the reaction product of a dialkylene glycol with a fatty acid or a fatty ester, to provide a lubricant composition;

10 b) containing said lubricant composition within a vessel.

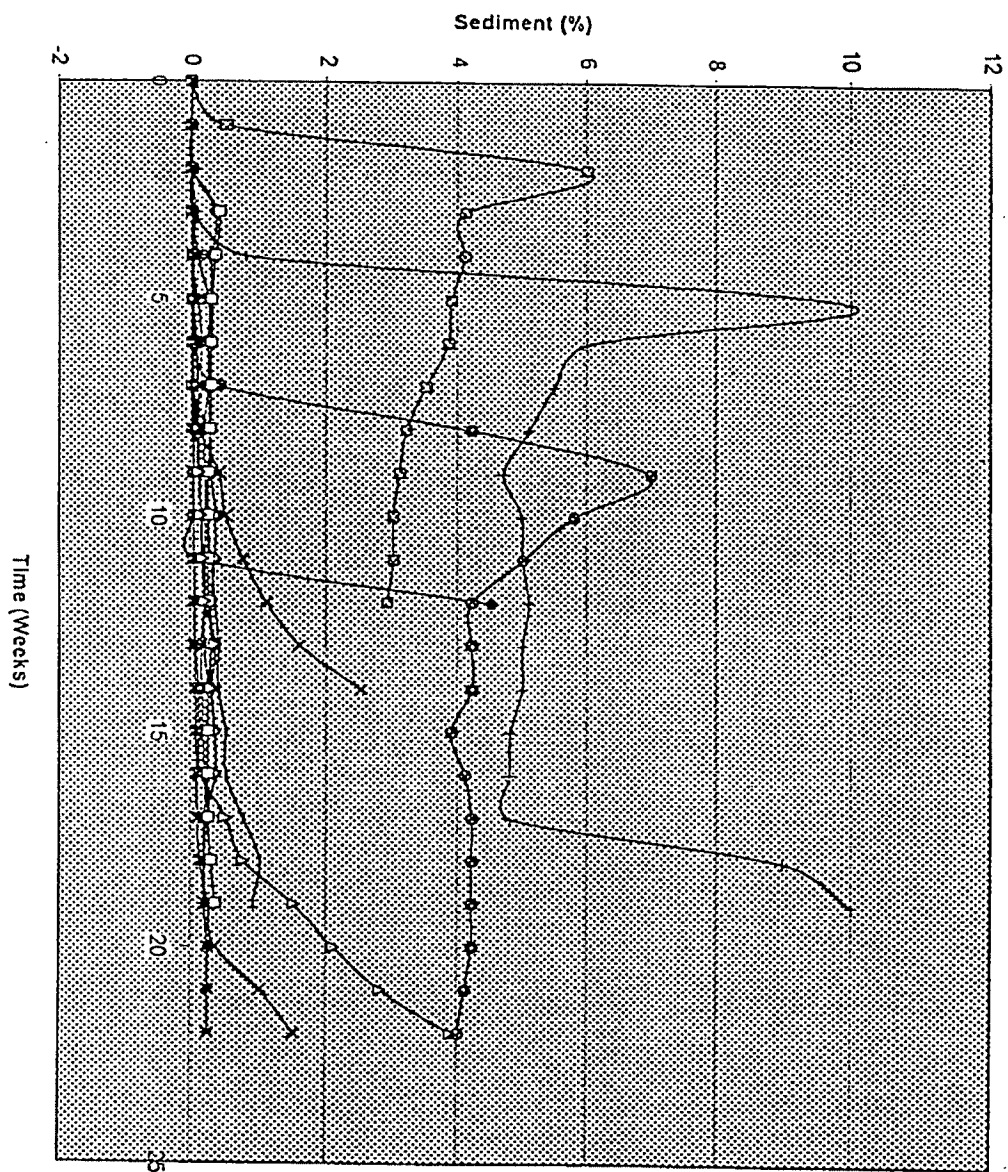
20. The method of claim 19 wherein said overbased alkaline earth sulfonate is overbased calcium sulfonate, and the friction modifier is selected from the group consisting of the reaction product of triethanolamine with one or more of a fatty compound selected from the group consisting of methyl oleate, tall oil fatty acid, oleic acid, isostearic acid and mixed oleic acid/stearic acid, overbased barium carboxylate, overbased calcium carboxylate and the reaction product of thiodiglycol with methyl  
20 oleate.

## ABSTRACT

An additive mixture for lubricant oils includes an overbased alkaline earth metal sulfonate, and at least one friction modifier selected from the group consisting of an overbased alkaline earth carboxylate, the reaction product of an alkanolamine with a fatty acid or a fatty acid ester and the reaction product of thiodiglycol or a dialkylene glycol with a fatty acid or a fatty acid ester. The additive mixture, when combined with a lubricant stock, provides a lubricant composition characterized by less sedimentation during storage.

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- TEA + methyl oleate
- TEA + TOFA
- \*— TEA + oleic acid
- \*— thiodiglycol + methyloleate
- diethyleneglycol + methyloleate
- +— TEA + ricinoleic acid
- +— TEA + isostearic acid
- +— TEA + erucic acid
- Alox 165L
- Uniflex 1803
- TEA + Century D-1
- \*— TEA + Century D1164

FIG. 1

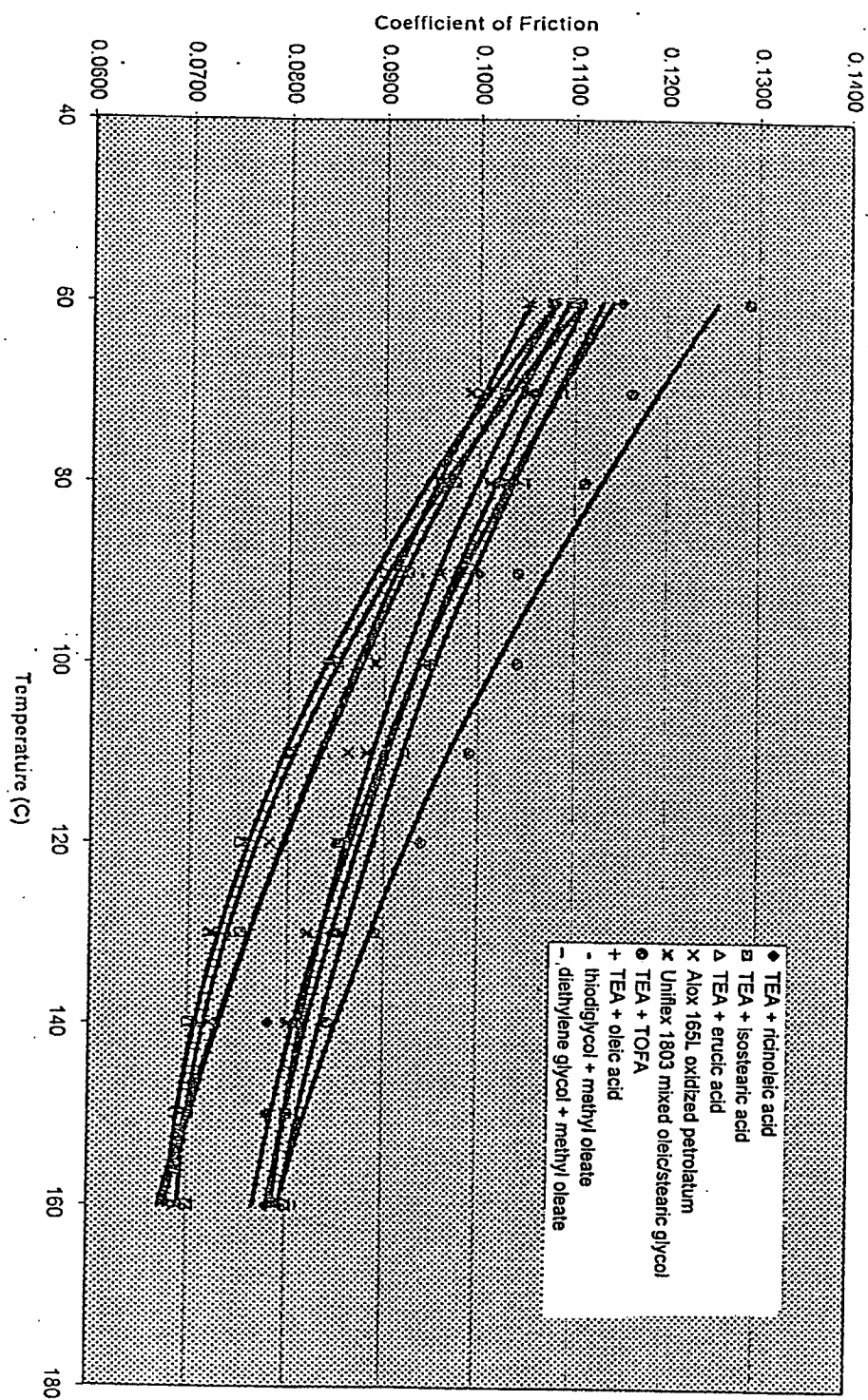


FIG. 2





COMBINED DECLARATION FOR PATENT  
DECLARATION AND POWER OF ATTORNEY

Atty Docket No. 0209-PA

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(Includes Reference to PCT International  
Applications)

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name.

I believe I am an original, first and joint inventor of the invention entitled:

**"LOW SEDIMENT FRICTION MODIFIERS"**

which is described and claimed in the patent specification which

( ) is attached hereto,

(X) was filed on DECEMBER 9, 2003 and accorded serial number 10/731,600, and for which  
invention Letters Patent are sought.

I hereby state that I have reviewed and understand the contents of the above identified  
specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to the examination of this  
application in accordance with Title 37, Code of Federal Regulations, Sec. 1.56(a).

I hereby claim foreign priority benefits under Title 35, United States Code, Sec. 119 of any  
foreign application(s) for patent or inventor's certificate listed below and have also identified below any  
foreign application for patent or inventor's certificate having a filing date before that of the application  
on which priority is claimed:

PRIOR FOREIGN/PCT APPLICATIONS AND PRIORITY CLAIMS UNDER 35 U.S.C. 119			
COUNTRY (if PCT, indicate "pct")	APPLICATION NO.	DATE OF FILING (month, day, year)	PRIORITY CLAIMED

COMBINED DECLARATION FOR PATENT  
APPLICATION AND POWER OF ATTORNEY

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(Includes Reference to PCT International  
Applications)

I hereby claim the benefit under Title 35, United States Code, Sec.120 of any United States application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of Title 35, United States Code, Sec.112, I acknowledge the duty to disclose material information as defined in Title 37, Code Federal Regulation Sec.1.56(a) which occurred between the filing date of the prior application and the national or PCT international filing date of this application:

PRIOR U.S. APPLICATIONS OR PCT INTERNATIONAL APPLICATIONS DESIGNATING THE U.S. FOR BENEFIT UNDER 35 U.S.C. 120					
U.S. APPLICATIONS			STATUS (CHECK ONE)		
US APPLN. NO.	US FILING DATE		PATENTED	PENDING	ABANDONED
PCT APPLICATIONS DESIGNATING THE U.S.					
PCT APPLICATION NO.	PCT FILING DATE	US SERIAL NOS. ASSIGNED (if any)			

I hereby appoint the following attorney's and/or agent(s) to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith: Kenneth D. Tremain - Reg. No. 20,518; and Daniel Reitenbach - Reg. No. 30,970 and Michael P. Dilworth - Reg. No. 37,311.

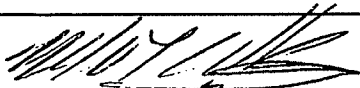
Address all correspondence to: Michael P. Dilworth Crompton Corporation Benson Road Middlebury, CT 06749	Direct telephone calls to:  Michael P. Dilworth tel. no.: (203) 573-3313
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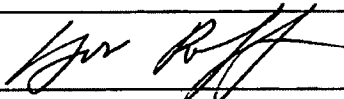
I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

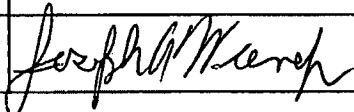
COMBINED DECLARATION FOR PATENT  
APPLICATION AND POWER OF ATTORNEY

Atty Docket No. 0209-PA  
Page 3 of 3

(Includes Reference to PCT International  
Applications)

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	SIGNATURE		DATE	1/5/04

**Amended Claims Currently Before The USPTO**

1.-11. (Canceled).

12. (Previously Presented) A lubricant composition comprising:

- a) a lubricant oil stock
- b) an amorphous overbased alkaline earth metal sulfonate in an amount sufficient to provide a sedimentation rate of no more than about 0.005% per week at 70C for at least 12 weeks; and,
- c) at least one friction modifier selected from the group consisting of a polyalkylene succinic anhydride, and overbased alkaline earth carboxylate, the reaction product of an alkanolamine with a fatty acid or a fatty acid ester, the reaction product of a thiodiglycol with a fatty acid or a fatty acid ester and the reaction product of a dialkylene glycol with a fatty acid or a fatty acid ester.

13. (Previously Presented) The lubricant composition of claim 12 wherein the overbased alkaline earth metal sulfonate is an amorphous overbased calcium sulfonate having a particle size of no more than about 30 nm.

14. (Previously Presented) The lubricant composition of claim 13 wherein the amorphous overbased calcium sulfonate has a TBN of at least about 400.

15. (Original) The lubricant composition of claim 12 wherein the friction modifier comprises the reaction product of a triethanolamine with a fatty acid or fatty acid ester.

16. (Previously Presented) The lubricant composition of claim 15 wherein the friction modifier comprises the reaction product of triethanolamine with one or more of a fatty compound selected from the group consisting of methyl oleate, tall oil fatty acid, oleic acid, ricinoleic acid, isostearic acid, erucic acid, mixed oleic acid/stearic acid and iso-oleic acid.

17. (Original) The lubricant composition of claim 12 wherein the friction modifier comprises the reaction product of thidiglycol with methyl oleate.

18. (Original) The lubricant composition of claim 12 wherein the friction modifier comprises the reaction product of diethylene glycol with methyl oleate.

19.-21. (Canceled).


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<p>1. Article Addressed to:</p> <p>Dr. Igor Riff            5 Colonial Court            Skillman, NJ 08558</p>		<p>B. Received by (Printed Name)            Igor Riff</p>	<p>C. Date of Delivery            05-11-09</p>
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102595-02-M-15

Justin Krieger – Katten, Washington, DC

cc: Jaimes Sher – Chemtura Corp., Middlebury, CT

Cc: David Kappos – USPTO, Alexandria, VA

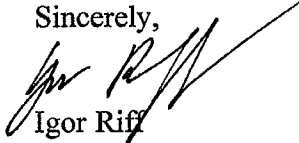
August 17, 2009

Dear Mr. Krieger:

In your letter from May 8, 2009 you have stated about your intention ``to correct the inventorship under the USPTO rules relating to uncooperative inventors``. I am not familiar with USPTO rules, and my understanding that your intention is to exclude my name as inventor for U.S. Patent Application No. 10/731,600 LOW SEDIMENT FRICTION MODIFIERS Chemtura Ref. 0209-PA.

My intention is to get familiar with this rule whether you have the rights to do so.

Sincerely,



Igor Riff